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Technology of spark plasma sintering as an innovative solution of synthesis high-density of SiC ceramics

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Abstract. The main advantages of spark plasma sintering (SPS) method compared to the classical hot pressing method for obtaining high-density ceramic based on silicon carbide are presented. Shows the perspective of obtaining a wide range of materials using this method. The possibilities of technology of spark plasma sintering of multicomponent ceramics based on silicon carbide of different composition have been studied. Established optimal SPS modes for sintering multicomponent ceramics of composition SiC (75%) - 22% (AlN) - Y₂O₃ (3%) (1800 °C/50MPa/15 min) provide formation of 100% dense composite, which allows to recommend these modes for making light and high-density ceramic material of specified composition with predicted properties.

The most widely used technology in the ceramic industry in recent years has been the method of spark plasma sintering due to the short duration of the process and energy efficiency. Using pulsed electric current and the so-called "spark plasma effect" ("plasma spark effect") is achieved very fast heating of the workpiece (up to 1000 ° C / min), so the sintering process is usually very short (several minutes). This allows you to suppress grain growth and obtain an equilibrium state, which opens up opportunities for creating new materials with previously unattainable compositions. In this work, we applied this method of spark plasma sintering (Spark Plasma Sintering, SPS), which is also known as the "Field Assisted Sintering Technology (FAST)", as an innovative solution for the synthesis of high-density ceramic based on silicon carbide.

It is known that sintering of ceramics based on silicon carbide with aluminum nitride by the classical technology of hot pressing at temperatures below 1900 ° C is undesirable due to the low diffusion rate of AlN in SiC at these temperatures. Sintering at temperatures above 2250 ° C leads to the decomposition of aluminum nitride. The optimal sintering time is 2-6 hours. With a sintering time of less than 2 hours, the formation of SiC-AlN solid solutions in the grain boundary region does not occur, which leads to a decrease in the strength and crack resistance of ceramics. With an increase in sintering time of more than 6 hours, diffusion processes between the backfill and sintered ceramics leads to embrittlement of the latter. The solution to these problems is seen in the application of the method of spark plasma sintering for this system.



The studied samples of SiC-AlN ceramics were obtained under various SPS-sintering modes, with the introduction of an additional sintering additive of yttrium oxide (Y_2O_3), which is a promoter of the sintering process, into the initial mixture of powders.

The objective of the research was to optimize the compositions of the mixtures of the starting components and the SPS modes to achieve the complex of the highest physical and mechanical properties (density, thermal conductivity, microhardness). The effect of the concentration of activating additives, temperature, and holding time in the SPS-sintering mode on the density of ceramics based on silicon carbide is estimated.

Based on the experience of many decades and the successful application of the classical technology of hot pressing, it was established [1] that the optimum sintering temperature of SiC-AlN-based ceramics, depending on the AlN content, is 2050-2300 K for 1 hour in an N₂ or Ar medium and a pressure of 10- 50 MPa. In early studies, the activating effect of yttrium oxide during sintering of ceramics based on SiC-AlN was studied [2-4]. When the content of yttrium oxide in the initial mixture is less than 2 wt.%, The final ceramic density does not exceed 90% TP, and an increase in the content of Y₂O₃ above 8 wt.% Leads to an increase in the oxidizability of the material due to the high diffusion mobility of yttrium ions at temperatures exceeding 1300 ° C. In addition, yttrium oxide is the most expensive component of the charge and an increase in its content leads to an increase in the cost of the material. We used this technique for SPS sintering of the compositions of the SiC-AlN system. Yttrium oxide was initially introduced into AlN powder, and then the corresponding mixtures with SiC were prepared. The starting components were mixed in the ratios shown in table 1.

Table 1. Composition and particle size of the powder (dispersion).

	Composition Components		
	SiC	AlN	Y ₂ O ₃
Weight % component.	75	25	0
	75	22	3
	75	18	7
Dispersion, microns	14.22	5.69	7.72

Granulometric analysis of the powders was carried out on a Fritsch Analysette 22 laser particle analyzer (Germany). The measuring range of the analyzer is 80 to 2000 nm. A fluid cell was used. Below are the results of the analysis of the particle size of the powder mixtures prepared for sintering (Figures 1 and 2).

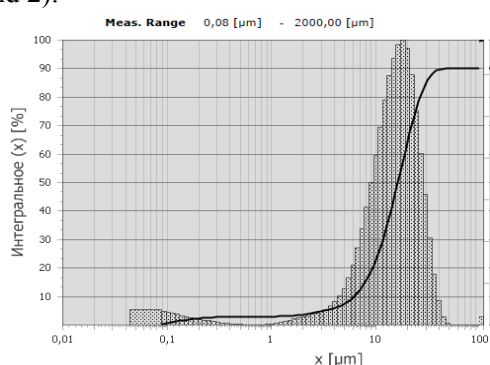


Figure 1. Particle size measurements using a laser analyzer for composition SiC (75%) - AlN (25%)

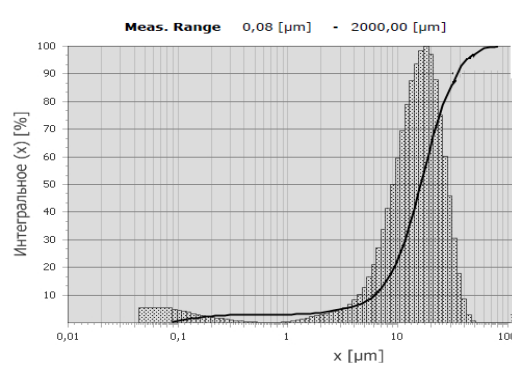


Figure 2. Particle size measurements using a laser analyzer for composition SiC (75%) - AlN (18%) - Y₂O₃ (7%)

Compositions after thorough mixing mainly contain a fraction of 8-14 microns.

The mixture of powders was molded and sintered by passing electric current through the billet in molds with an inner diameter of 24 mm made of isostatic graphite I-3 (area 4.52 cm²), using an additional layer of graphite paper between the matrix and powder filling and between graphite punches and powder

to prevent the reaction between the powder and the tool material. Download - 10g. Temperature control was carried out by an optical pyrometer. Sintering was carried out by applying constant pressure to the powder throughout the entire time. The samples were cooled freely in the chamber.

Using the well-known and previously described technological methods [5, 6], experimental batches of ceramic samples of various compositions were obtained (Figure 3). Figure 3 clearly shows how the color changes, and therefore the density of the obtained samples, depending on the concentration of the starting components and sintering conditions (temperature, time).

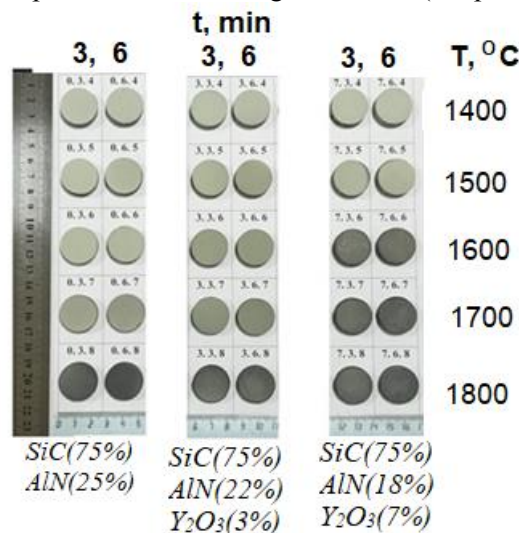


Figure 3. Ceramic samples obtained by the SPS method of various compositions under different sintering conditions.

The resulting samples were removed from the mold, cleaned and tested for density. It was found that the addition of yttrium oxide significantly increases the density of SiC-AlN-based ceramics. SPS sintering at a temperature of 1800 ° C of finely dispersed mixtures of SiC and AlN with the addition of yttrium oxide already at a content of 18 and 22% AlN provides a relative density of 0.95 and 0.98, respectively, which is significantly higher than the density value for ceramics obtained by hot pressing. (Figures 4 and 5).

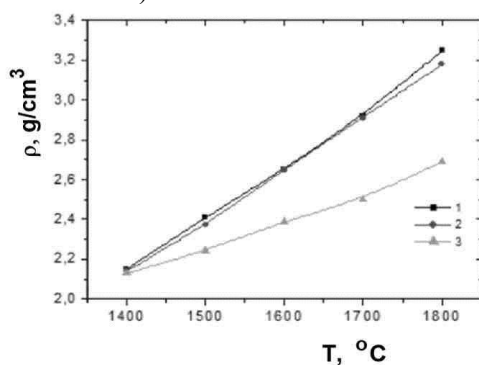


Figure 4. Temperature dependence of density of ceramic samples of various composition 1.SiC(75%)-AlN(18%)-Y₂O₃(7%), 2.SiC(75%)-AlN(22%)-Y₂O₃(3%), 3.SiC(75%)-AlN(25%). Sintering time - 3min.

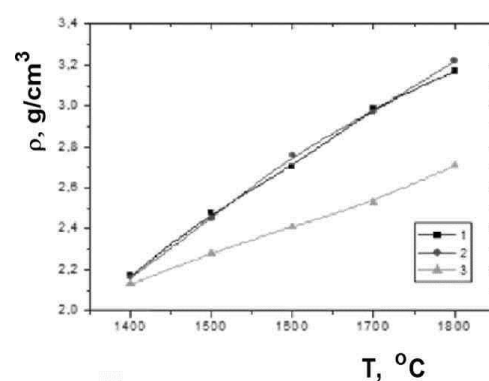


Figure 5. Temperature dependence of density of ceramic samples of various composition 1.SiC(75%)-AlN(18%)-Y₂O₃(7%), 2.SiC(75%)-AlN(22%)-Y₂O₃(3%), 3.SiC(75%)-AlN(25%). Sintering time - 6min.

During the experiment, it was found that upon sintering of samples with the addition of 7% yttrium oxide at 1800 ° C in the mold, the formation of a liquid phase is observed, which fills the space between the particles of silicon carbide until a dense material is formed. This is confirmed by the microstructure

photographs shown in Figures 6 and 7, obtained on a field emission SEM with a nanolithography unit, Raith150TWO (Germany).

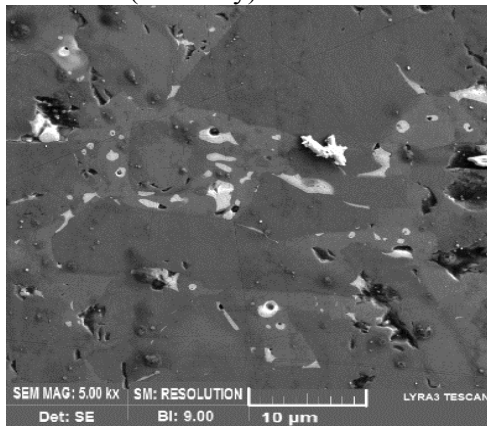


Figure 6. SEM image of the structure of ceramic SiC(75%)-AlN(18%)-Y₂O₃(7%) obtained by the SPS method.

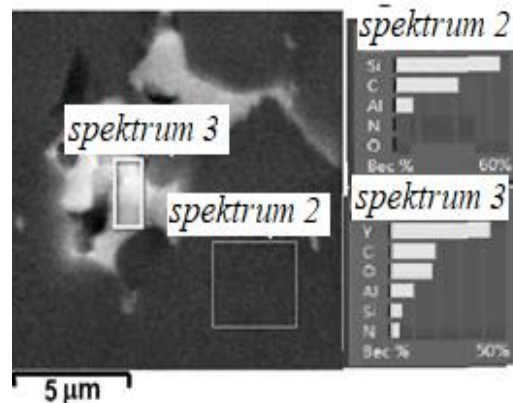


Figure 7. Results of elemental analysis of ceramic SiC(75%)-AlN(18%)-Y₂O₃(7%) obtained by the SPS method.

An increase in the concentration of activating additive leads to an increase in the evaporation of oxides and an increase in their interaction with silicon carbide, which reduces the density of the SiC material. At a high content of oxides ($\geq 7\%$ wt.), They fill the pore space, and the excess liquid melt is squeezed out of the sintered sample, corrodes the protective graphite foil and interacts with the die matrix. The oxide melt adheres to the graphite form and destroys the sample.

Thus, for SiC-AlN ceramics, the optimal additive is Y₂O₃ - 7 wt.% At a sintering temperature of 1800 °C. Therefore, further studies were conducted on the effect of the exposure time on the sintering mode at a maximum sintering temperature of 1800 °C on the density of the obtained samples for only two compositions: 0% and 3% yttrium oxide. Research results for density are shown in Figure 8.

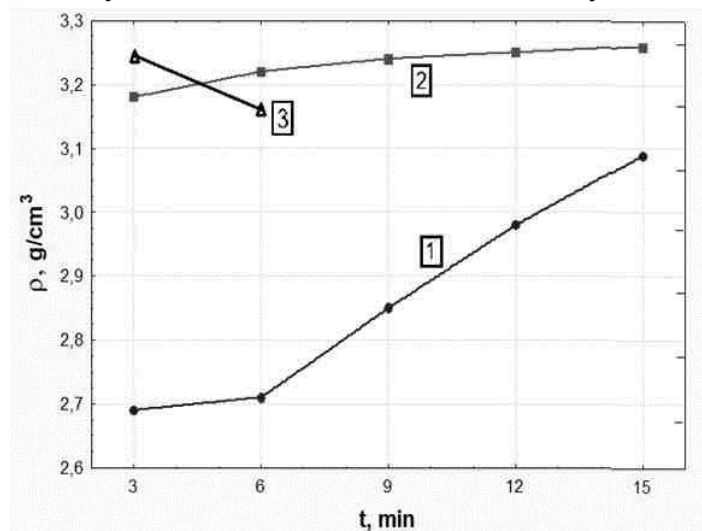


Figure 8. The dependence of the density of ceramic samples on the sintering time in the regime at T = 1800 °C: 1.SiC(75%)-AlN(25%). 2.SiC(75%)-AlN(22%)-Y₂O₃(3%). 3.SiC(75%)-AlN(18%)-Y₂O₃(7%).

It was found that the highest density value of ~ 95.9% of the theoretical value for the SiC (75%) - AlN (25%) composition is achieved by SPS sintering at a temperature of T = 1800 OC for a time t = 15 minutes. And with the addition of 3% yttrium oxide at the same technological parameters, 100% of the theoretical density is achieved.

The presented results show that a high-density silicon carbide based ceramic with perfect microstructure and high (up to 100%) density can be obtained using the plasma-micron method from a micron fraction powder, which opens up prospects for the widespread use of the SPS method for the economical production of high-quality ceramic products from silicon carbide of a given composition with predictable properties.

The research results are useful for optimizing the manufacturing process of structural and functional high-density ceramics. Using the available data, you can find the optimal parameters of the sintering process to obtain certain properties. The dependences presented in the work will help to evaluate the influence of plasma-spark sintering parameters on the structure and properties of the obtained ceramic materials. Studies of these promising results are ongoing, and experiments are being conducted with finely divided raw materials.

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